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ABSTRACT

We report on research results in this project which synergize advanced material science approaches with fundamental optical physics concepts pertaining to light-matter interaction, with the goal of solving seminal problems for the development of very high performance light emitting diodes (LEDs) in the blue and near ultraviolet for Solid State Lighting applications. Accomplishments in the second 12 month contract period include (i) new means of synthesizing AlGaIn and InN quantum dots by droplet heteroepitaxy, (ii) synthesis of AlGaIn nanowires as building blocks for GaN-based microcavity devices, (iii) progress towards direct epitaxial alignment of the dense arrays of nanowires, (iv) observation and measurements of stimulated emission in dense InGaIn nanopost arrays, (v) design and fabrication of InGaIn photonic crystal emitters, and (vi) observation and measurements of enhanced fluorescence from coupled quantum dot and plasmonic nanostructures. The body of results is presented in this report shows how a solid foundation has been laid, with several noticeable accomplishments, for innovative research, consistent with the stated milestones.

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EXECUTIVE SUMMARY

The aim of this research project at Brown and Yale Universities is to synergize advanced material science approaches with fundamental optical physics concepts pertaining to light-matter interaction, with the goal of solving fundamental efficiency problems so as to enable the development of very high performance light emitting diodes (LEDs) in the blue and near ultraviolet for Solid State Lighting applications, covering the spectral regime of approximately 370-480 nm. Our work is directed towards novel, highly adaptable device concepts that enable their flexible utilization and matching to the broad spectrum of approaches and requirements that pertain to contemporary solid state lighting approaches. The material base of the light emitters is formed from nanostructured gallium nitride and related semiconductor heterostructures, which are enclosed by mesoscopic optical confinement and light emitting enclosures for efficient extraction of optical energy. The ultimate goal of the research is to reach the goal of a highly wall-plug efficient, high optical power device by concentrating on two specific, closely coupled performance dictating elements within the LED.

The research program has been organized in the following way: First, in ongoing work we are synthesizing nanostructured active media to enhance the internal radiative efficiency utilizing special concepts in epitaxial growth for growing quantum dots and quantum wires. Second, the research has also focused focus on the design and fabrication of advanced photonic confinement structures, which encase the nanostructured active medium for enhancing the spontaneous emission by strengthening light-matter interaction at a fundamental level and for efficiently extracting and distributing the photons for delivery into specific geometrical radiation patterns by design.

Below we summarize the progress made during the second year of the project. The body of results is presented clearly indicates that a solid foundation has been laid, in addition to several noticeable accomplishments, for reaching the stated milestones in the coming years. Specifically we have made substantially advances in the synthesis of zero- and one-dimensional GaN nanostructures, established the building blocks for making GaN-based microcavity devices, and demonstrated a top-down approach to nano-scale photonic devices for enhanced spontaneous emission and light extraction.

I. INTRODUCTION

A revolution in lighting regarding energy saving, component lifetime, and fixture versatility is anticipated if the traditional tube-based (incandescent and fluorescent) lamps can be replaced by solid state LEDs. For this vision to become reality, a substantial gap in terms of light output between a standard incandescent bulb (1000~2000 lumens) and a single III-N LED chip (1~5 lumens) needs to be bridged. Engineering issues such as metal contacts, current spreading, and light extraction have to be addressed in scaling up the device area for increased optical output from a single chip. Of paramount importance is the identification and realization of an active medium capable of more efficient conversion of electron-hole pairs into photons, especially under higher-level current injection ($J=10^3\sim10^4$ A/cm²). Carrier localization due to In-related compositional fluctuations in InGa_N is attributed to the constraint of in-plane carrier diffusion, thus preserving and preventing the injected carriers from recombining at nonradiative dislocation sites. Such a benefit of localization diminishes at high-level injection as more carriers acquire a nature of extended electronic states and are consumed by nonradiative processes. A recent observation of a substantial decay in quantum efficiency at a modest injection level from state-of-the-art high power AlGaInN LEDs¹ served as a reminder of the necessity of innovative and creative material research for an efficient light-emitting medium.

In this report we summarize the progress made during the second year of the DOE Contract entitled “Nanostructured High Performance Ultraviolet and Blue Light Emitting Diodes for Solid State Lighting”. Details of the experimental facilities and methodology are described in experimental section (II). The body of results is presented in Section III which clearly indicated that a solid foundation has been laid, in addition to several noticeable accomplishments, for reaching the stated milestones in the coming years. Specifically we have made substantially advances in the synthesis of zero- and one-dimensional GaN nanostructures, established the building blocks for making GaN-based microcavity devices, and demonstrated a top-down approach to nano-scale photonic devices for enhanced spontaneous emission and light extraction.

II. EXPERIMENTAL

MOCVD growth was carried out in a horizontal reactor (Aixtron 200/4 HT-S) using trimethylgallium (TMGa), trimethylaluminum (TMAI), and ammonia (NH₃) as sources with hydrogen and nitrogen as carrier gases. Atomic force microscopy (AFM) was conducted using a Digital Instrument Nanoprobe III model with tapping mode. PL signal was collected using a 30 ns pulsed frequency-quadrupled solid laser emitting at 263 nm. Scanning electron microscopy (SEM) was performed using a LEO 1530 Field Emission SEM. Chemical composition is determined by energy dispersive x-ray spectroscopy (EDS) that is embedded in the SEM. High-resolution transmission electron microscopy (TEM) and electron diffraction (ED) were performed using a FEI Tecnai 20 Field Emission TEM. Scanning energy dispersive x-ray spectrometry (EDS) was carried out in a Philips CM-12 TEM with a nominal beam diameter of 50 nm.

III. RESULTS AND DISCUSSION

III.A Beyond GaN quantum dots: AlGaIn and InN quantum dots by droplet heteroepitaxy

Near and above its melting point, metallic thin film transforms into nanoscale liquid droplets, a phenomenon that does not require the presence of strain mismatch or surfactant. The formation of metal droplets due to surface tension presents an alternative route to semiconductor nanostructures by converting the droplets into crystalline QDs. Koguchi et al.² has demonstrated the formation of nanometer Ga droplets on GaAs substrates and the conversion into GaAs QDs through an exposure to arsenic vapor. Kawasaki et al.³ and Hu et al.⁴ have reported the preparation of GaN QDs on, respectively, AlGaIn and SiC by droplet conversion using gas source MBE. Only low temperature luminescence was observed in these samples; a deviation of microscopic stoichiometry was identified as a factor compromising the recombination efficiency. Last year we demonstrated that gallium droplet heteroepitaxy (DHE) is a valid path toward the flexible synthesis of GaN QDs with high optical efficacy. The formation of Ga droplets, including the nucleation dynamics and kinetics, had been examined at the atomic scale. The conversion of gallium droplets upon exposure to ammonia proceeds with two competing mechanisms, a liquid-phase-epitaxy-like crystallization of quantum dots and the diffusion-based two-dimensional growth of GaN layers, both can be regulated by surface kinetics. Photoluminescence (PL) at 345 nm at room temperature suggests that the converted QDs are optically active and can be a candidate for ultraviolet emitters.

III.A.1 AlGaIn quantum dots

This year we extended our DHE study to the alloy system of AlGaInN. In the formation of AlGaIn quantum dots, we first mixed in the gas phase the Ga and Al

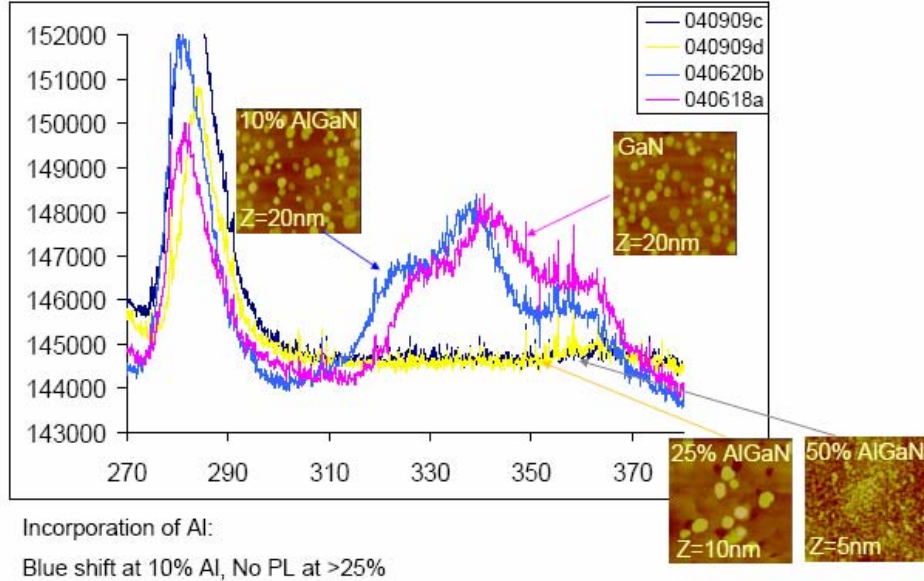


Fig 1. Room temperature PL from GaN and AlGaIn quantum dots with varying Al compositions.

precursors and create metallic droplets on AlGaIn surface.

Conversion of Al/Ga nanodroplets into AlGaN quantum dots was preformed by exposing the Al/Ga droplets to ammonia flow immediately after the interruption of TMAI and TMGa flow, followed by a rapid cooling of the samples under NH_3 flow. The conversion process represents interplay between several competing mechanisms. The first is the reaction of nitrogen with Al/Ga nanodroplets, resulting in the formation of a supersaturated solution and consequently the crystallization of AlN/GaN at the droplet/AlGaN interface. On the other hand, the transition from nanodroplets to crystal can proceed differently when Ga and Al adatoms have a high surface diffusion rate; the diminishing role of the surface tension as Al and Ga droplets solidify in the absence of a high compressive strain makes it possible for the Al and Ga atoms to diffuse out from the droplets and participate in a layer-by-layer growth.⁵ It was discovered that, as the Al composition in the droplets increases above 40%, the conversion process proceeds with the formation of dispersive small grains intermixed with layered nucleation.

High resolution electron beam lithography has been used to create photonic crystal and sub-wavelength nanostructures to investigate the local electromagnetic interactions on the nanoscale.

Optical properties of GaN obtained by DHE were investigated by absorption, PL, and near field techniques. Additional advanced methods have involved time-resolved luminescence measurements.

III.A.2 InN quantum dots

Toward solid-state lighting in the longer wavelength region of the visible spectrum, we have begun in March 2005 the synthesis of In-rich InGaN nanostructures. Our device plan is to incorporate In(Ga)N-based nanostructures into the active junction region of a light-emitting diode to take advantages of both enhanced confinement and suppressed non-radiative recombination. In the month of April we began the synthesis of InN nanowires as well as InN quantum dots. AFM images captured at various stages of dot formation are shown in Fig 2. Optical investigation is in progress and will be reported later.

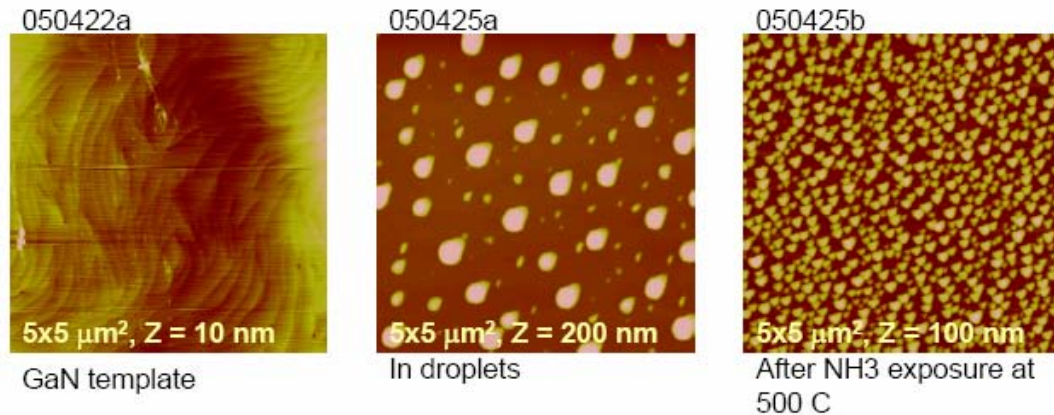


Fig 2. AFM images of (left) a GaN epilayer, (center) metallic indium droplets prepared at 500 C, and (right) InN quantum dots after NH_3 exposure and conversion.

To In conclusion, it has been demonstrated that DHE of AlGa on conductive AlGaIn templates and InN on GaN is a viable path towards visible light emission incorporating zero-dimensional structures in the active region. Optically active AlGaIn-capped QDs exhibiting PL emission at 330 nm at room temperature were obtained by stabilizing the density of the AlGaIn islands throughout the process of DHE and subsequent annealing.

III.B. Synthesis of AlGaIn Nanowires

To date the synthesis of GaN nanowires are performed primarily by a near-equilibrium, tube furnace technique which does not offer optimum control or flexibility. In this letter we report our observations in preparing III-N nanowires using a conventional MOCVD system. In addition to demonstrating GaN and AlN nanowires from a cold-wall, commercial platform, which has not been reported to our knowledge, we outline the critical issues of nanowire synthesis and present specific solutions. The versatility of using MOCVD for nanowire synthesis is illustrated by the fabrication of three-dimensional (3D) GaN/AlN nano-trees.

The basic principle of VLS mechanism for anisotropic growth of 1D nanostructures was summarized by Wagner.⁴ Prerequisites that were identified include: (1) A sizable disparity in reaction kinetics between regular vapor-solid (VS) and the VLS mechanisms, thus mandating a low supersaturation for growth selectivity; (2) the creation and retention of liquid droplets to facilitate adsorption and incorporation of vapor phase species; and (3) the need to have nucleation sites with appropriate crystallographic orientations conducive to the minimization of surface energies. Criterion (1) helps to elucidate the popularity and success in the synthesis of GaN nanowires through hot-wall, flow-tube furnace chamber in which elemental Ga source is placed upstream of catalyst-treated substrates.⁶ The proximity of a desorptive source to an adjacent growth surface spontaneously creates an ambient that is close to thermodynamic equilibrium. Favorable conditions for nanowires growth (under low supersaturation) is empirically derived by adjusting the relative positioning between the Ga boat and substrate due to spatial gradients in temperature and Ga flux. On the other hand, modern MOCVD involves a much different and complex process in which organometallic precursors (undersaturated even at room temperature) are transported in vapor phase with minimum dissociation to the vicinity of growth surface. The precursors then undergo rapid pyrolysis decomposition upon entering the heated zone near surface (thermal boundary layer), creating a highly inhomogeneous profile and a mass-transport limited growth process.⁷

During the first year of the current DOE project, we have demonstrated the synthesis of GaN nanowires by MOCVD and provided very detailed structural analysis (Fig. 3). In the 2nd year of this DOE project we carried out the synthesis of alloy nanowires and explored the possibility of aligning the nanowires through crystallographic epitaxy in order to incorporate these nanowires into conventional light emitting devices.

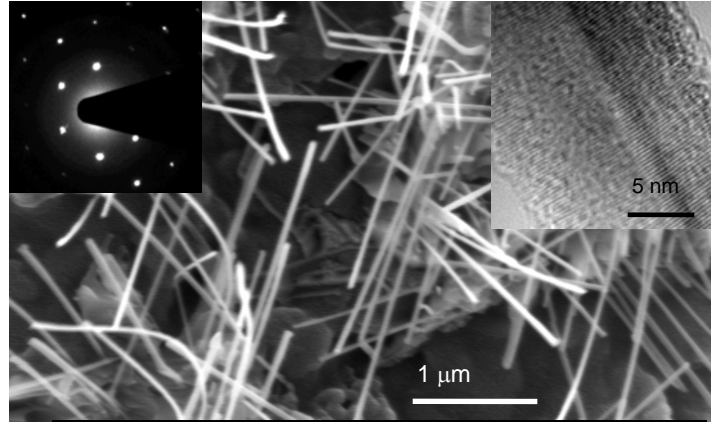


Figure 3. SEM of GaN:In nanowires on MCM-41 templates. Inset: High resolution TEM (right) and electron diffraction images (left)

III.B.1 Synthesis of AlGaN nanowires

The morphology of MOCVD-grown AlGaN nanowires with a varying vapor-phase Al content, $x_{Al-gas} = [TMAI]/([TMAI]+[TMGa])$, is surveyed by SEM and summarized in Fig 1. Nanowires are observed in samples with Al vapor content from 30 to 90%. As the Al concentration is increased, an inverse dependence is observed between the length of the wires and the density of the nanowires/nanocrystals. Samples with a high x_{Al-gas} exhibit a clean background and a low density of long (4-5 μm) nanowires while samples with low x_{Al-gas} are characterized by short, rod-like (1-2 μm) nanowires with nanocrystals decorating the background. Quantitative analysis of such morphological trends is hindered by a random, haystack distribution of nanowires on alumina substrates; details of the analysis of epitaxially aligned AlGaN wires on crystalline templates will be reported elsewhere. Under the same flow conditions with the same metalorganic flow rate, growth of GaN results in a mixture of short nanorods and nanocrystals while growth of AlN leads to a thin film coating that conforms to the morphology of alumina substrates under SEM imaging.

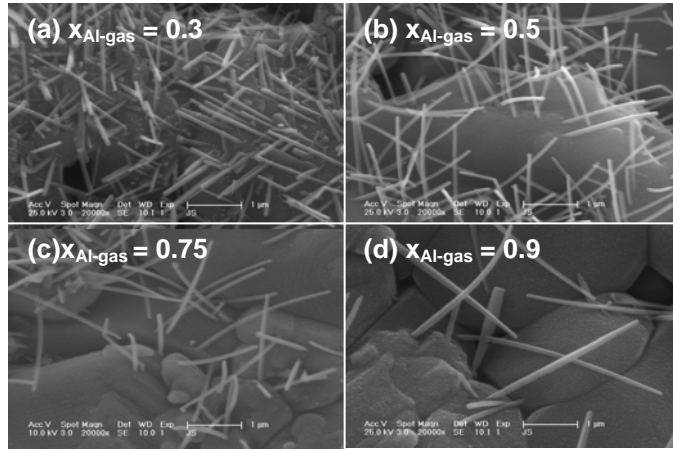


Fig 4. SEM images of AlGaIn nanowires on alumina templates with Al gas phase ratio $x_{\text{Al-gas}} = 30\%$, 50% , 75% , and 90%

Microstructural analysis was carried out on nanowires that had been sonicated in solvent solution and dispersed onto TEM grids; a typical example is shown in Fig 2a. A contrast of dark inner core and light outer sheath is observed for all the AlGaIn nanowires imaged. Thickness and the degree of tapering of the outer sheath region increases with the increase of $x_{\text{Al-gas}}$; a slope of $\sim 11 \text{ nm}/\mu\text{m}$ is estimated for the 90% sample. Chemical analysis by selective area EDS, with an electron-beam cross-section of 3 nm in diameter, was performed at five different spots over the width of the nanowires. Normalized composition profiles based on Al and Ga line peak intensity ratios are shown in Fig 2b. The image contrast in Fig 2a correlates well with the composition profile (Fig. 2b) according to a designation of an Al-rich sheath region and a core consisting of primarily GaN. Our observation of spontaneous formation of coaxial AlGaIn nanostructures resembles an earlier finding⁸ of synthesis of AlGaIn nanowires by a hot-wall furnace reactor in which a lattice mismatched strain was proposed as a driving force responsible for the formation of spatially segregated coaxial nanowires.

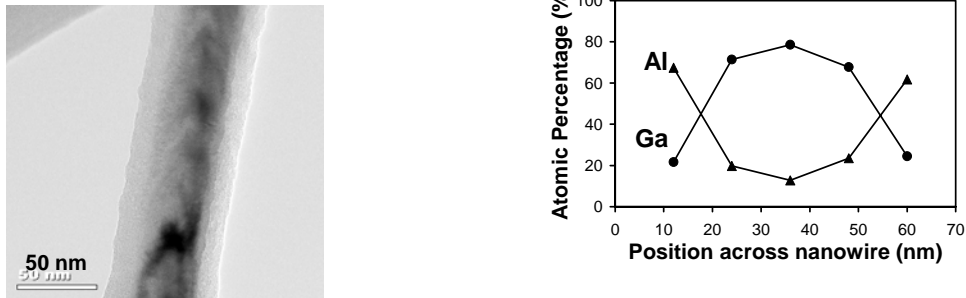


Figure 5. (a) TEM of AlGaIn nanowires with $x_{\text{Al-gas}} = 50\%$, (b) Normalized composition profile across the nanowire. Composition profile across the nanowire, the horizontal axis is the position across the nanowire, vertical axis is the atomic percentage of Al (triangular), Ga (round), and N (square).

Nanowire synthesis based on near-equilibrium vapor-liquid-solid (VLS) principle in flow-tube environment is a well-documented procedure.⁹ Growth selectivity is derived from a preferential incorporation of vapor species through or around catalyst nanodroplets and a negligible growth rate at the vapor-solid interface imposed by minimum supersaturation near equilibrium. In the synthesis of nanowires using non-equilibrium, epitaxial techniques such as MOCVD and MBE, surface kinetics including adatom diffusion need to be taken into consideration.¹⁰ It was shown recently¹¹ that growth of InAs nanowires using CBE takes place largely through the preferential incorporation at the droplet tip of adatoms that are within the radius of the diffusion length. In our attempt of synthesizing AlGaIn nanowires by MOCVD, presence of both Al and Ga adatoms, as well a large difference in bond strength and consequently diffusion mobility between the two species,¹² creates a unique interplay between kinetic and thermodynamic processes which will be elaborated in the following.

Fig 3a shows a high resolution TEM image near the tip of a nanowire oriented in the [101-0] direction. For all the nanowire tips examined, the width of the core (GaIn) region as determined by the contrast is well correlated with the physical dimension of catalyst droplets, suggesting strongly that the preferential incorporation into and the formation of GaIn core region is linked to the catalytic growth. The presence of Al-rich Al(Ga)In outer sheath that is located outside the shadow projection of nanodroplets is unlikely related to catalytic growth. Observations of the absence in lateral over growth of AlGaIn¹³ and little surface diffusion of Al adatoms lend credibility to a model that the AlGaIn outer sheath is deposited through conventional vapor-solid growth with negligible selectivity. Such a designation provides an explanation of the increased tapering of AlGaIn nanowires with an increasing x_{Al-gas} since the tapering slope represents an indicator of the prevalence of sidewall growth. We note that the dimensional matching between catalyst nanodroplets and the core diameter of coaxial nanowires has been reported in the intentional overgrowth of InGaIn shell on pre-grown GaIn nanowires.¹⁴

High-resolution TEM also reveals the presence of well-defined crystallographic planes at the nanowire tips that can be categorized into inclined (Fig. 3a) and pointed (not shown) for [101-0] and [112-0] nanowires, respectively. Crystallographic analysis on both types nanowires indicate the droplet/nanowire interfaces in both cases correspond to pyramidal [101-1] plane, a plane that is likely to have the lowest surface energy or growth rate during the MOCVD growth of GaIn.¹⁵ Based on the electron diffraction data, crystallographic symmetry, and ELO works, a three-dimensional reconstruction of the tip structure is given in Fig. 3b. Shape evolution in nucleation and nanocrystal growth has been analyzed by Wulff theorem in terms of surface energy and adopted to the kinetic consideration of growth rates.¹⁶ A well-known phenomenon is that a rapidly growing surface (or thermodynamically a crystal facet with high surface energy) tends to grow itself into extinction. Our consideration of surface energetics (Fig 3b), on the contrary, leads to an intriguing if not paradoxical distinction for nanowire growth in which selective and preferential growth, mediated by the presence of catalyst and/or liquid droplets, takes place at and is confined to a surface/interface with a low surface energy,¹⁷ thus forming a self-sustaining growth process.

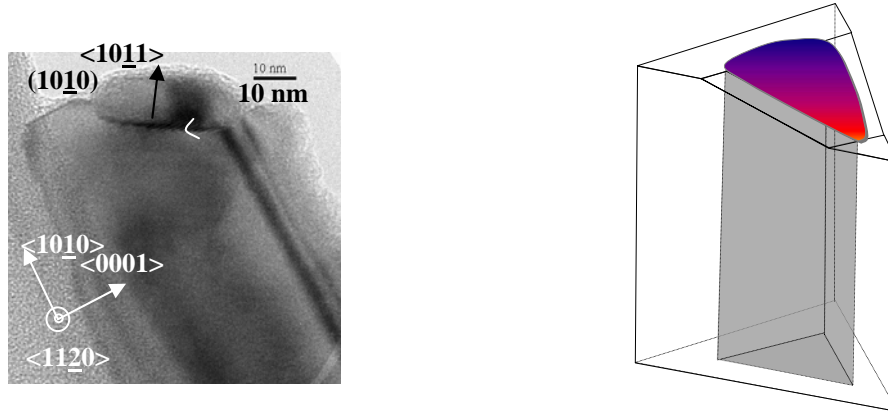


Figure 6. (a) HRTEM of AlGaIn ($x_{\text{Al-gas}} = 50\%$) nanowires near the tip region, the nanowires growth direction is along $\langle 10\bar{1}0 \rangle$ and the view angle is parallel to $(10\bar{1}0)$ plane and along $\langle 11\bar{2}0 \rangle$ direction, with a very small degree of tilting toward $\langle 0001 \rangle$ or $\langle 000\bar{1} \rangle$ direction, (b) 3D diagram of the TEM image, the triangular cross section is bound by two $\{11\bar{2}2\}$ planes and basal (0001) plane, the droplet is on the tilted $(10\bar{1}1)$ plane.

To ascertain both the optical quality and the spatial distribution of the alloyed nanostructures, cathodoluminescence was performed on two samples with $x_{\text{Al-gas}} = 50\%$ and 70% . Fig 4a shows wide-area ($10 \times 10 \mu\text{m}^2$) integrated scanning CL spectra in which emission from both samples are dominated by a broadened GaN band-to-band recombination peak at 370nm at 105 K. A broad shoulder on the high-energy side (~ 320 nm) is also observed in both samples. Origin of different emission signatures is investigated through monochromatic scanning CL mapping at a wavelength of 370 nm (Fig 4c) and 320 nm (Fig 4d), with SEM imaging recorded for spatial reference (Fig 4b). A point-to-point match between the nanowire features under SEM (Fig. 4b) and the spatially resolved emission pattern at 370 nm (Fig. 4c) unambiguously associates the dominant GaN emission to the individual nanowires, presumably from the core region of the coaxial AlGaIn nanowires. Furthermore, CL mapping at 320 nm reveals a nearly homogeneous distribution of AlGaIn, consistent with the notion that, under the growth condition employed, Al has a relatively low surface mobility.

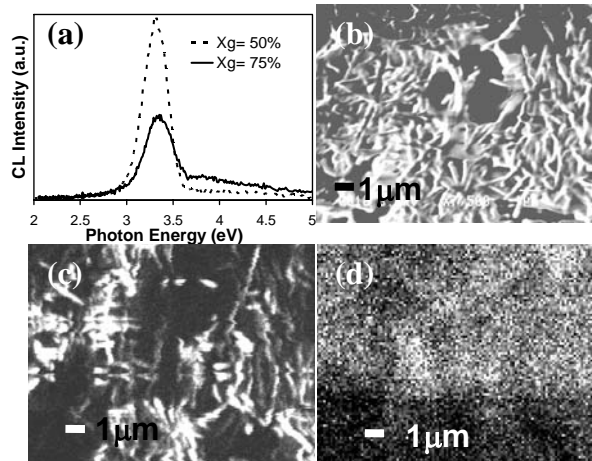


Figure 7. (a) Wide area ($10 \times 10 \mu\text{m}^2$) integrated scanning CL spectra at 105 K for AlGaIn nanowires with $x_{\text{Al-gas}} = 50\%$ and 75% , (b) SEM image of AlGaIn nanowires with $x_{\text{Al-gas}} = 75\%$ on alumina substrate, (c) monochromatic CL map of the same area at wavelengths of 370 nm (3.35 eV), (d) monochromatic CL map of the same area at 320 nm (3.87 eV). The scale bar is 1 μm . The white area corresponds to the position where CL intensity is strong.

Non-equilibrium growth processes such MOCVD and MBE have been instrumental in preparing ternary and quaternary semiconductors in planar epitaxy. Attempts to produce nano-planar, patterned growth of alloys often results in complex composition profiles due to disparate and competitive kinetic properties among the constituting elements. The spontaneous formation of coaxial AlGaIn nanowires through conventional procedure of precursor mixing in a MOCVD process attests to the complicate nature of non-equilibrium growth. In addition to the catalytic growth of GaN core and the V-S deposition of AlN sheath, we note that the concurrent presence of Al adatoms (or AlN) leads to a surprising phenomenon of enhanced surface diffusion of Ga adatoms, as can be inferred from Fig 1 with a reducing density and increasing length of nanowires as $x_{\text{Al-gas}}$ increases. This hypothesis is corroborated by a report that AlN is effective as a mask in epitaxial lateral over growth,¹⁸ indicating that the diffusion of Ga adatoms on AlN (or high Al containing alloy) is greatly enhanced. It has been pointed out that engineering of surface energy play a crucial role in controlling the shape and morphology of nanostructures.¹⁹ In the synthesis of AlGaIn nanowires, it is speculated that the passivation of the GaN core by the AlN sheath helps to stabilize the sidewall facets and improve the nanowire viability at nucleation stage, bearing the same root as earlier reports using hydrogen or oxide.

III.B.2 Synthesis of InGaIn coaxial nanowires

Incorporation of indium into GaN-based nanowires is one of the milestones in shifting the wavelength of III-nitride nanostructures into the visible spectrum for solid-state lighting. Given one fundamental difficulty in avoiding phase separation through the VLS-mode of growth, and the disparity in surface diffusion based on our experience in the growth of AlGaIn nanowires (Section III.B.1), we switched to a hybrid mode of synthesis, using VLS-grown AlGaIn nanowires as the nanoscale crystalline templates. InGaIn layers are grown epitaxially along the radially direction (orange region) and a cylindrical quantum wells. Figure 8 shows a schematic drawing of the coaxial structure

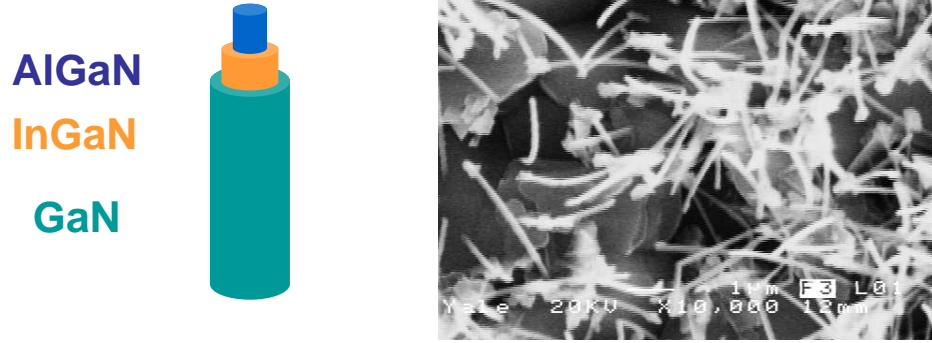


Figure 8. (Left) Schematic drawing of a heterostructure coaxial nanowire, (Right) SEM of AlGaIn/InGaIn/GaN coaxial nanowires with InGaIn cylindrical quantum wells.

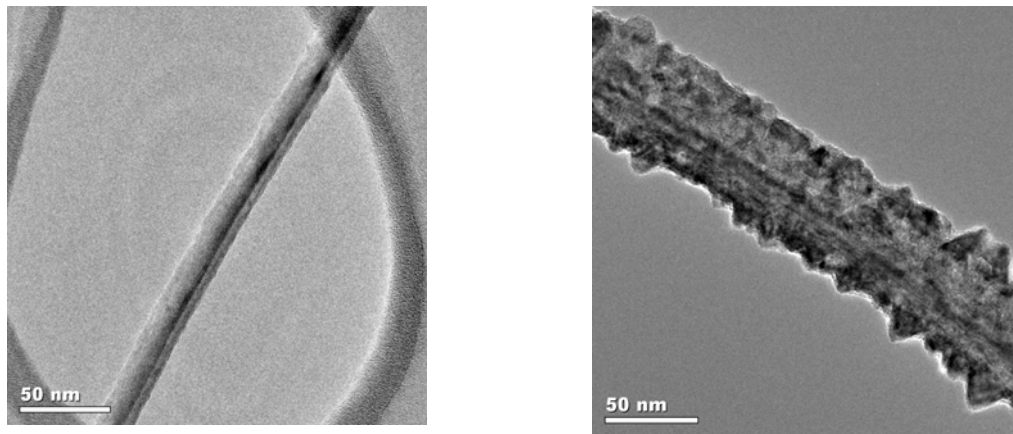


Figure 9. (Left) TEM of an AlGaIn wire that is the core of the structure, and (right) TEM of an GaN/InGaIn/AlGaIn composite coaxial nanowires

(left) and the SEM morphology of these composite nanowires. TEM study shows (Figure 9) that the AlGaIn nanowires have relatively smooth side surface at a scale of 50 nm. Close examination did reveal, however, rough surface crystallites with a roughness fluctuation on the order of 5 nm. Once we switch the growth to the conventional vapor-solid mode, InGaIn and GaN growth tends to amplify the pre-existing roughness on AlGaIn and caused the formation of small islands with height of about 20 nm. Growth of InGaIn at reduced temperature is known to cause surface roughening and the formation of V-defects. The observed roughening is perhaps strain driven and due to low surface diffusion at our low growth temperature.

Optical characterization is key to our understanding of the electronic properties of

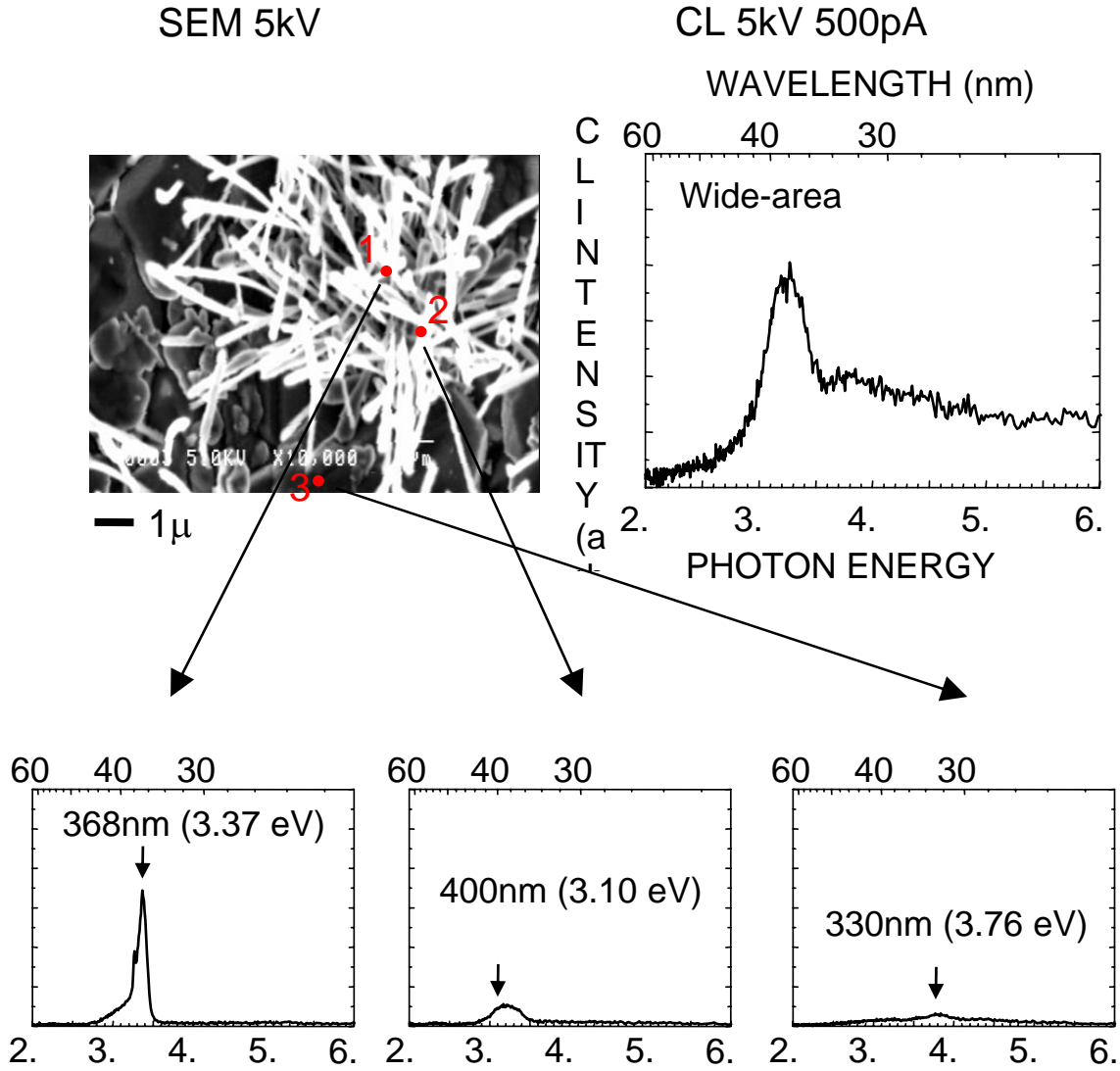


Fig 10. (Upper left) SEM image of a InGaN coaxial-nanowire sample, (upper right) wide-area CL emission spectrum, (lower panels) CL spectrum (spot excitation) from points 1, 2, and 3.

nanostructures. Cathodoluminescence is employed to probe the optical property with SEM-level spatial resolution. We have seen areas emitting at from GaN nanowire trunks (point 1), InGaN emission at 400 nm (point 2), and from a non-selective coating of AlGaIn (point 3). The use of nanowires as nano-substrates could change the growth behavior of InGaIn and perhaps lead to better quality InGaIn at a high indium content.

III.B.3 Synthesis of InN nanowires

We have also attempted the growth of InN nanowires using near-equilibrium growth procedure. Growth of InN represents a delicate balance among three competing forces: the formation of indium droplets (condensation), the etching of In/InN (desorption), and the growth of InN (crystallization). SEM images (Fig 11, upper left and right) show the formation of InN nanowires with a diameter of 100 nm and a length of up to 6 μm.

Selective-area EDS indicates (lower right panel) that the nanowires consist of mostly InN. Optical characterization is being performed.

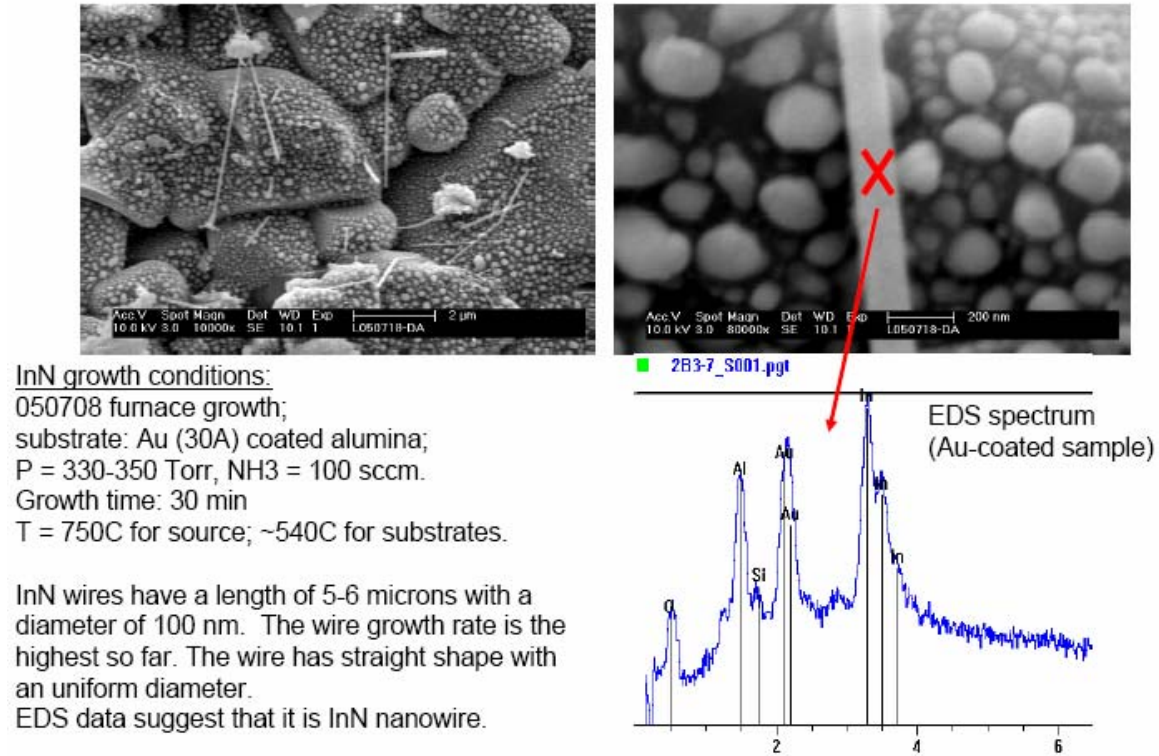


Fig 11. SEM images of InN nanowires with a background of spherical-shape In droplets. High resolution EDS confirms the chemical constitution of the nanowires to be InN.

III.B.4 Epitaxial alignment of nanowires

Crystallographic information forms the basis in designing and selecting an epitaxial system. It is identified from electron diffraction that MOCVD nanowires examined in this work exhibit $[10\bar{1}0]$ growth directions independent of the templates, in contrast with some of the reported data.²⁰ It is likely that, under the particular Ga-rich VLS growth mode, the $\{10\bar{1}0\}$ planes emerge as the lowest-energy ones with the formation of Ga-Ga dimer reconstruction.²¹ Selected area electron diffraction and SEM further indicate that our MOCVD grown nanowires have a triangular cross-section, bound by $\{0001\}$ and two $\{11\bar{2}2\}$ planes. Concerning the cross section of GaN nanowires, a useful analogy can be drawn from shape evolution during GaN epitaxial lateral overgrowth (ELO).²² $(000\bar{1})$, $(11\bar{2}0)$, and $(11\bar{2}2)$ have been identified as the surfaces that have low densities of dangling bonds (surface reconstruction is ignored) and in principle can be made free of atomic steps. The particular triangular cross section resembles triangular mesas when the ELO stripes are aligned along the $\langle 10\bar{1}0 \rangle$ directions.²³

Growth of GaN epilayers along non-polar directions including M-axis is of contemporary interests due to the possibility of achieving heterostructures that are free of built-in electric fields.^{24,25} To test the idea of crystallographic alignment of nanowires epitaxially, GaN nanowires were grown on crystalline templates including C-plane GaN (0001) epilayers, M-plane AlN substrate (Crystal IS, Inc), and alumina substrates as a

reference. Growth on alumina exhibited typical haystack morphology (Fig 12a) owing to the random nature of the crystallographic orientation of the nucleation sites. Only crystallites were observed on C-plane GaN (0001) epilayer, in support of the assertion that VLS growth mode is closely related to crystallographic match and not crystalline perfection. Lastly, the growth on M-plane AlN substrates produces vertically aligned GaN nanowires having a triangular cross section (Fig. 12b). The triangular cross section is consistent with the designations from electron diffraction studies. While the majority of nanowires grow vertically, we also observe nanowires that are inclined 60° to surface normal along the equivalent $\langle 10\bar{1}0 \rangle$ M-axes. In regions with a reduced coverage of catalyst metal (e-beam evaporated nickel), the density of nanowires is reduced accordingly (inset). In regions without nickel coating, only faceted crystallites of sub-micron size were found. With a combination of lithography and self-assembled patterning, vertical growth of ordered III-N nanowires on M-plane AlN or GaN substrates will facilitate practical implementation of nano-devices such as nanowire LEDs and field emitter displays.²⁶

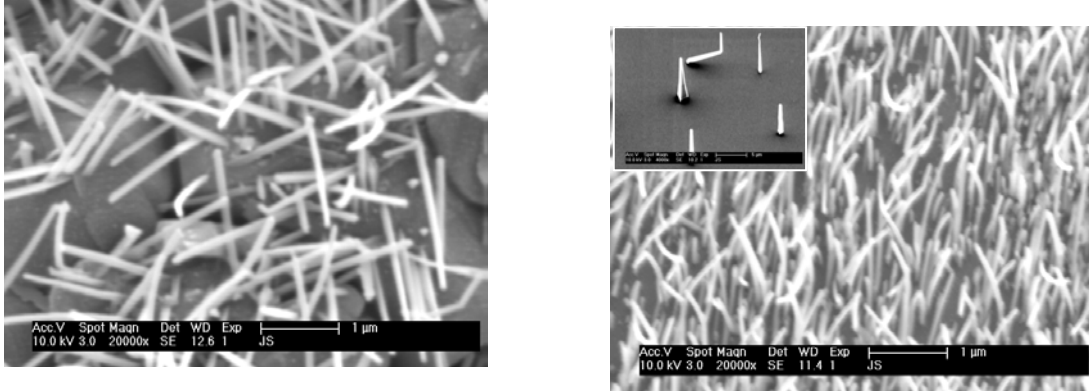


Fig 12. SEM images of GaN nanowires on (a) alumina substrates and (b) M-plane AlN substrates.

The concept of selective epitaxial growth of GaN nanowires is further extended into templates with more complex topography. It is known that GaN nucleation on sapphire substrates, under either non-optimum conditions or before the complete coalescence, can at times exhibit a rough morphology with discrete C-axis oriented GaN mesas. These mesas typically possess a hexagonal symmetry with flat plateaus of (0001) basal plane (confirmed by x-ray diffraction) and pyramidal sidewalls. The employment of faceted GaN mesas enables a combinatorial survey of the tendency and selectivity of nanowire growth along different planes. Figure 13 shows plane-view SEM of AlGaIn nanowires grown on such a “rough” GaN template. Horizontally-aligned nanowires emanating from the $\{10\bar{1}0\}$ M-planes, forming directional arrays of nanowires of comparable diameters and lengths, can be clearly seen. The (0001) C-plane, labeled C in Fig. 13a, is found not conducive to the nucleation of III-N nanowires, only crystallites were observed on top of the flat plateau. As pyramidal $\{10\bar{1}n\}$ M-planes assume more C-plane character with an increasing n , the efficacy of supporting nanowire growth diminishes (M' planes in Fig. 13a). While the prism A-plane $\{11\bar{2}0\}$ is not observed for the GaN mesas examined, the fact that pyramidal $\{11\bar{2}n\}$ planes (labeled A') remain clean while neighboring pyramidal $\{10\bar{1}n\}$ planes of a comparable degree of inclination exhibit a high density of nanowires

implies a substantial and useful selectivity, due to disparity in atomic configuration and surface energy, between the M- and A-planes. The demonstration of horizontal M-axis nanowires presents a unique opportunity of integrating non-polar nanowire-based LEDs into C-plane GaN structures. Fig. 14 shows, in a proof-of-concept way, the plausibility of interconnecting two GaN mesas with an array of M-axis GaN nanowires, in which the number/density and location of the nanowires can be tailored conceivably by lithographical patterning of catalyst thin films. In contrast to the current paradigm of post-synthesis dispersion and manipulation of nanowires, a new class of horizontal nanowire devices interconnected epitaxially between contact mesas, as well as complex nanowire networks, can be envisioned through a combination of lithography, epitaxial lateral overgrowth, and crystallographically aligned VLS growth.

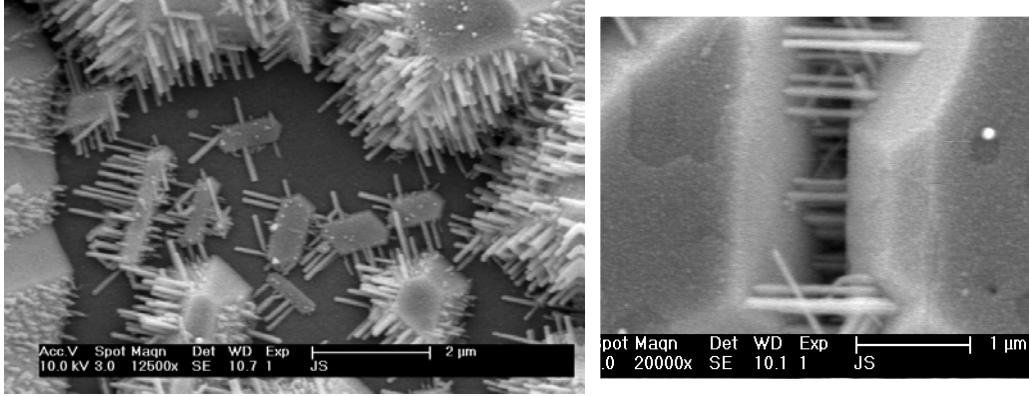


Fig 13. SEM of GaN horizontal nanowires emanating from hexagonal islands. Fig 14. GaN nanowires interconnect.

Typically MOCVD growth is carried out under diffusion-limited condition. This mechanism is manifested in a systematic variation of nanowire morphology from the edge of the sample²⁷ toward center. A series of SEM images were taken progressively from sample edge (“0 μm ” in Fig 15) toward the center of wafer. Horizontal nanowires along the (1010) directions are observed in all cases, even though the length and density of nanowires exhibit monotonic variations. The profile of exponential-like decay in the length of nanowires over hundreds of micrometers resembles what was observed in the selective area growth of GaN from the edge of a half-plane dielectric mask²⁸ and was attributed to lateral (parallel to surface) diffusion in the gas phase due to a step-like change in the concentration of metalorganic precursors across the wafer (or mask) edge. A higher concentration of TMGa near the wafer edge is responsible for longer nanowires. However, the observation of increasingly high density of wires toward the center and the appearance of nuclei on top of the GaN mesas cannot be adequately explained by the concentration gradient in gas phase alone.

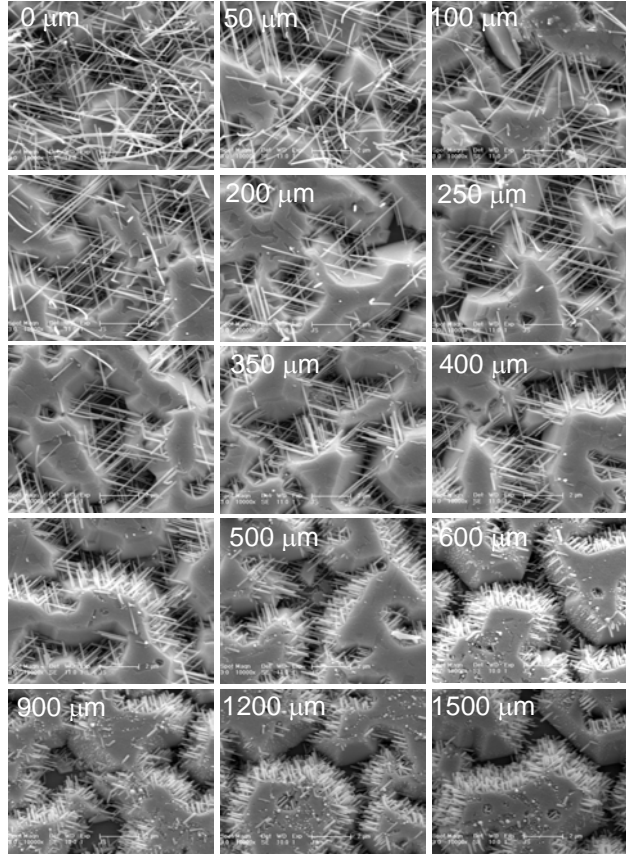


Figure 15. Plane-view SEM image of GaN nanowires on a “rough” GaN template on sapphire, refer to text for details.

III.C. Light emission from dense InGaN nanopost arrays

We have used so-called top-down approach to fabricate dense GaN nano-posts. First, the III-nitride heterostructure was grown by organometallic vapor phase epitaxy (OMVPE) on (0001) sapphire substrate. As shown in figure 16, following the growth of a GaN buffer layer and n-type GaN layer, ten undoped InGaN quantum well was grown as active medium designed to emits around 400 nm light. The epitaxy growth is ended with a ~20nm p-type doped top layer. This layer is designed so thin to facility the dry etching.

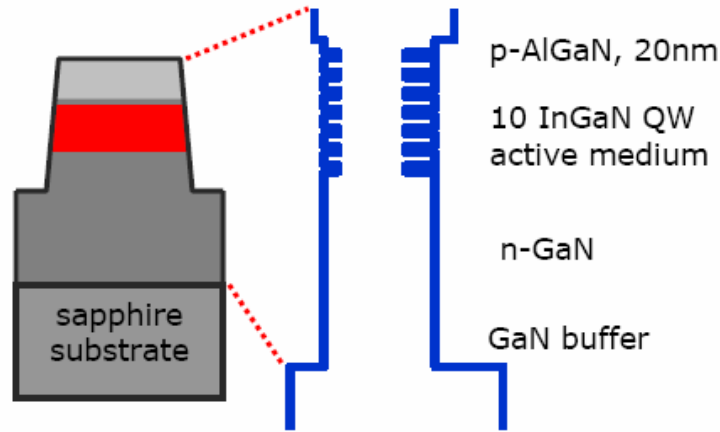
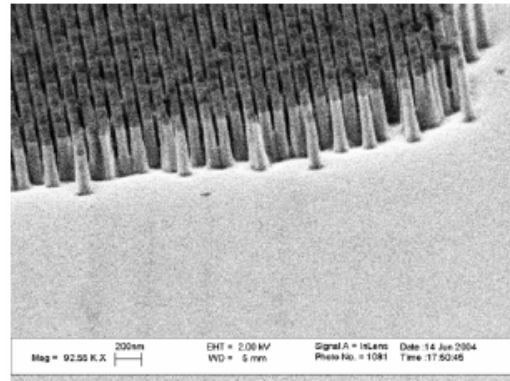
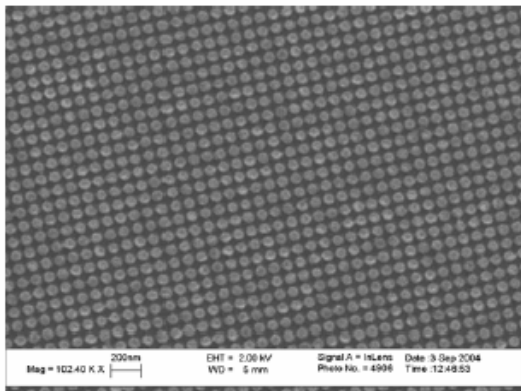


Fig 16. Schematics of a InGaN MQW p-n junction used in this study.

Several different architectures of arrays have been fabricated through e-beam lithography generated pattern and dry etching pattern transfer techniques. These patterns include 1D linear arrays, 2D square lattice arrays, and 2D hexangular arrays with different post diameters and lattice constants. Figure 17 shows SEM images of some examples, from both top view and side view.

Robust spontaneous light emissions have been observed in the photoluminescence (PL) experiment. We have focused the pumping laser (frequency tripled 355nm Nd:YAG pulsed laser) vertically onto the front surface and imaging the backside emitted light to a spectrometer. We found that the PL efficiency of the patterned nanopost array is comparable to that of the unpatterned sample. Another confirmation is the carrier lifetime measurement. The combination of relative PL efficiency and lifetime measurements suggests that radiative recombination remains the dominant process for the e-h pairs in our patterned nanopost samples. There could be two reasons that nonradiative recombination at free surfaces of GaN and its alloys does not dominate electron-hole pair dynamics to the same degree of severity as with ‘conventional’ III-V semiconductors. The first is the innate ability of the nitride surfaces to reconstruct so as to remove the surface Fermi level from the bandgap. The short e-h diffusion lengths due to carrier localization in InGaN QWs (from pronounced alloy potential fluctuations) could be the reason of very high radiative efficiency.

Stimulated emission has also been observed from these structures. Apart from residual photonic crystal-type effects, another reason for the stimulated emission could be



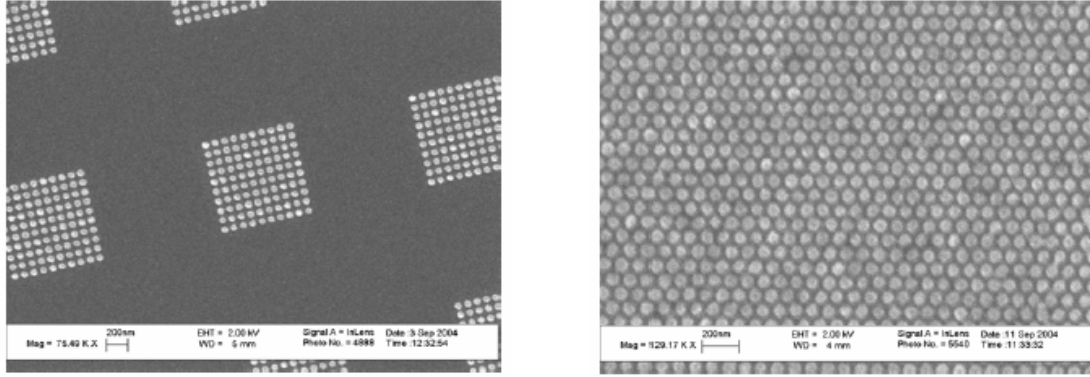


Fig 17. Examples of lithographically-defined InGaN nano-post

the near field interaction between the nearest individual nano-size light emitters. Our nano-posts are so dense that the separation is below 30nm. In principle, the emission cross section of individual nano-post could be enhanced with such a close separation. We have measured the carrier life-time at different pumping levels in spontaneous regime. As Figure 18 shows, the carrier life-time decrease at higher pumping level. This result is controversial to a typical result for spontaneous emission, where the carrier life-time would increase due to the saturation of nonradiative recombination at high pumping level.

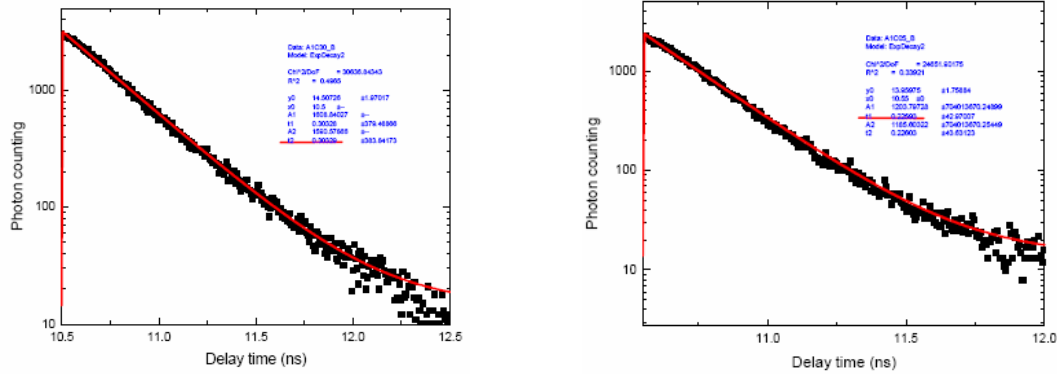


Fig 18 Carrier life-time of nano-posts at low (left, 303 ps) and high (right, 226 ps) pumping levels.

III.D. Analysis of Nitride Photonic Crystal Structures

In Section III.C we addressed experimental observation of enhanced and stimulated optically pumped blue emission from unique InGaN quantum well nanostructures, formed from planar LED-type wafers by electron beam lithography. The process resulted in ~60-90nm diameter nitride rods, densely arrayed with ~30 nm edge-to-edge inter-rod separation. We consider here possible photonic bandgap contributions to the observed low-threshold stimulated emission, formed due the periodic distribution of the refractive index. We have used simulation based on two dimension finite difference time domain approach (FDTD) to calculate the photonic bandgap element of our nanopost arrays. The adjacent Figure 1 shows an example of the simulations of the photonic crystal structure, while Figure 2 displays the corresponding reflection spectrum in the spectral range of interest. We chose the input parameters as the follows: the GaN nano-post refractive index is 2.54, the nano-post diameter is 70nm, and the period of the nano-posts is 90nm. There is only a very narrow photonic bandgap near the QW emission (400nm), which means the

photonic bandgap effect could be very weak. The reflection simulation is done with a launch of light from the edge of the nano-posts (2D), and the result shows that QW emission is at the edge of the photonic bandgap, i.e. at edge of the wavelength range where the light propagation is forbidden. It is also over what wavelength range of the photons that are confined and therefore feedback is provided. We need to point out the parameter values for inputs to the simulation are only the best guess based on our

knowledge. For example, the exact number of nano-posts' refractive index at high pumping levels is unknown. Another uncertainty is the diameter of GaN nano-posts. It is very hard to measure it accurately via SEM image due to the well-known charging effect in SEM. Unfortunately, our simulations show that the position of photonic bandgap is very sensitive to those inputs. So far, our experimental results could not totally rule out photonic bandgap lasing possibility. However, we need to point out that a difference between our device and a typical photonic bandgap laser is that our heterostructures do not have a designed waveguide for lateral propagation.

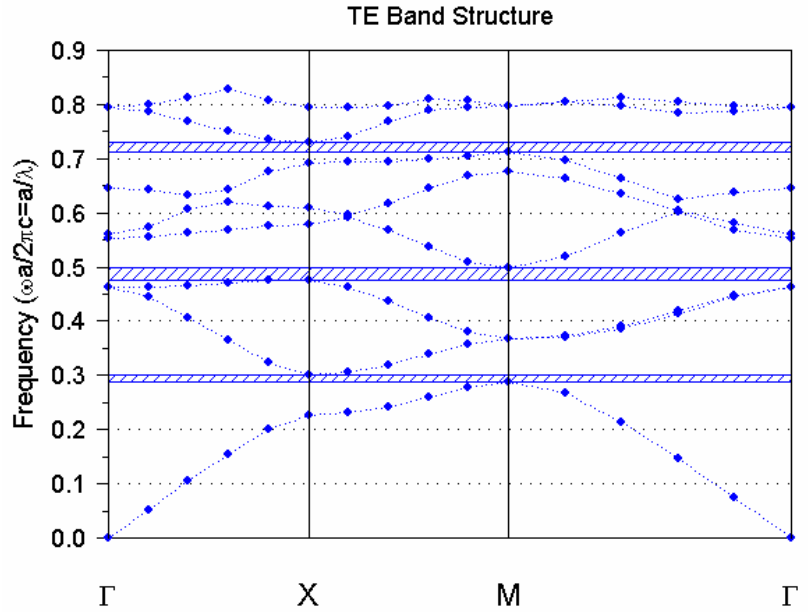
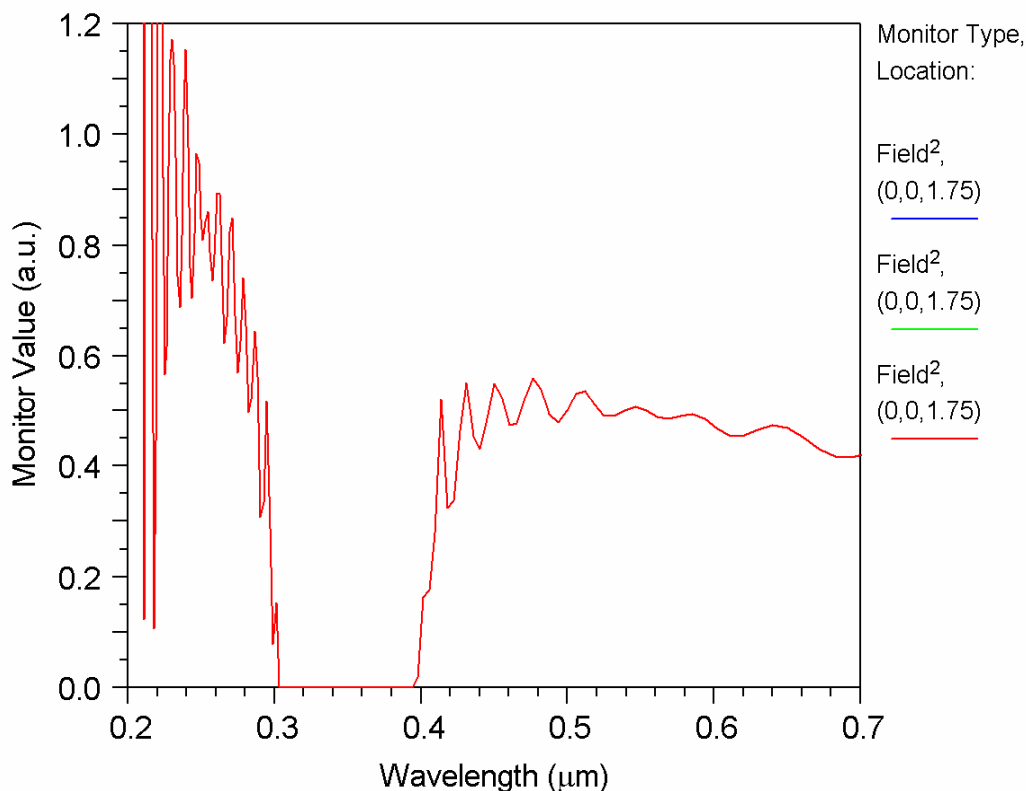


Figure 19: Example of FDTD simulation of the 2D photonic crystal structure for an array of 70 nm diameter InGaN QW nanoposts

If the effect of photonic bandgap is indeed small, the stimulated emission we have observed could be the result of near field interaction between the nearest individual nano-size light emitters since, our nano-posts are so dense that the separation is below 30nm. In principle, the emission cross section of individual nano-post could be enhanced with such a close separation. To further study this effect, we have measured the carrier life-time at different pumping levels in spontaneous regime. We have observed that the carrier life-time decrease at higher pumping level. This result is opposite to a typical



result for spontaneous emission, where the carrier life-time would increase due to the saturation of nonradiative recombination at high pumping level.

At this point, we cannot not totally rule out the contribution by photonic bandgap effect to stimulated emission. However, the unusual optical effects seen in the laboratory, especially the spatially localized stimulated emission and carrier life-time results suggest that the stimulated emission is different from the stimulated emission which occurs in the presence of feedback from optical resonators.

III.E. Near optical field interaction between semiconductor and metal nanoparticles

Presence of noble metal nanoparticles can significantly impact the spontaneous emission from fluorescent nanoparticles located within the surface plasmon field of metal. Excitation of surface plasmons in the metal nanoparticles can yield several orders of

magnitude stronger local electromagnetic field than the average incident optical field. However, plasmon induced modification of fluorescence always involves the competition between possible enhancement and demonstrated severe quenching, the latter due to nonradiative energy transfer from the luminescent species to the metal. Thus careful design of proper material architecture with nanoscale control of the spatial relationship between the fluorescent and metallic nanoparticles is called for to achieve optimal emission enhancement. Many differing reports have appeared in chemical physics studies of fluorescent dye molecules attached to metal surfaces. Here we report on engineered nanostructures where colloidal semiconductor nanocrystal quantum dots (QDs) are selectively deposited on designed plasmonically active templates. Colloidal semiconductor QDs themselves have been the subject of many scientific and practical studies because of their excellent optical properties in visible region and ability to adapt to novel applications, such as a biological labels and solution processed solar cells [2]. To control and to minimize non-radiative quenching in the coupled QD-plasmon system, which is most pronounced when the fluorescent species are in direct contact with the metal particles [1], we prepared QDs dispersed in PMMA thin film (50nm) and created planar Au- and Ag-particle arrays using such doped PMMA films as the resist for electron-beam lithography. The interparticle edge-to-edge distances of the 2D arrays were kept short ($< 160\text{nm}$) so as to have most of QDs impacted by the localized plasmon field.

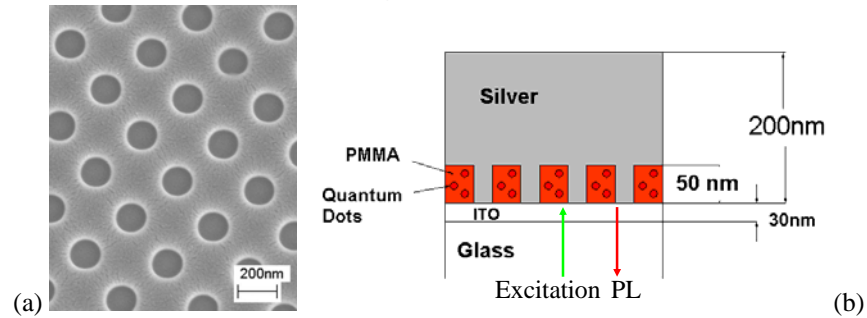


Fig 21. (a) SEM image of square array of holes in PMMA after developing. (Thin Au/Pd film was sputtered to avoid the charging effect in imaging). The diameter of the particles is 160 nm and the lattice constant is 300 nm. (b) Schematic of typical finished sample structure. Fluorescence is excited by a 532 nm laser line through and detected from the glass substrate side at normal incident angle.

The sample fabrication involve colloidal CdSe/ZnS core-shell quantum dots with emission wavelength centered at 650nm which were dissolved in chloroform and mixed with a polymethyl methacrylate (PMMA) 2% solution in chlorobenzene, typically with the ratio of 1:1. The QD/PMMA solution was then spin-coated on an ITO coated glass slide, and baked at 180°C for 6 minutes to make a 50nm thin film. No macroscopic aggregation was observed under fluorescence microscope. After generating the periodic array patterns by e-beam lithography and developing, a 200nm thick Ag film was deposited on the substrate. A scanning electron microscopy image of a typical array of holes after developing the PMMA is shown in Fig. 21(a).

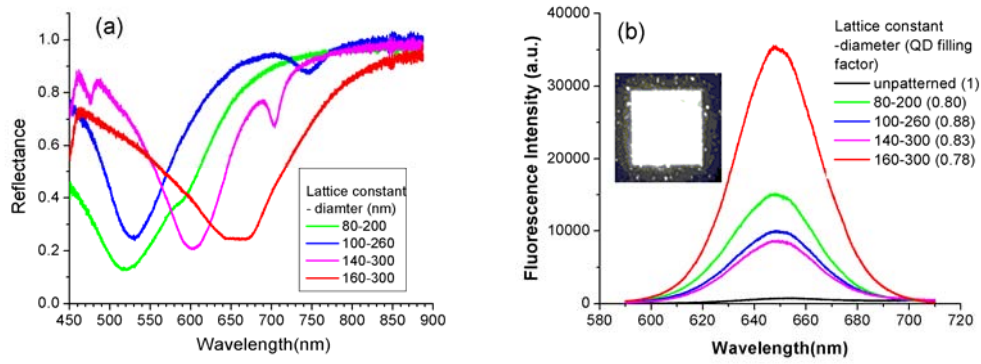


Fig 22. Reflectance (a) and fluorescence (b) spectra of various QD-plasmon patterns. Dimensions and QD filling factor is indicated in (b). Inset in (b) shows a far field fluorescence microscopy image from the pattern “80-200” where lesser QD fluorescence yield from outside the plasmonic pattern appears dark in this image.

Fig 22 (a) shows an example of how the plasmon resonance of the Ag-QD patterns is tunable from $\sim 500\text{nm}$ to $\sim 650\text{nm}$ by the plasmon template size. Fluorescence spectroscopy of the nanocomposite was performed using 532 nm laser line as an excitation source in a standard microspectroscopy system. As shown in Fig 22 (b), all of plasmon templates show enhancement of their QD-derived fluorescence, but the strongest enhancement (\sim factor of 50) occurs when the plasmon energy is tuned to resonance with the exciton emission energy of QDs. As a reference, we also measured the fluorescence from an unpatterned QD-doped PMMA thin film with and without Ag layers atop, in order to check the effect by the unpatterned Ag layer. The fluorescence from “bare” QD-doped PMMA film was quite weak, whereas the presence of a uniform Ag-coat did improve the fluorescence intensity somewhat due to reflection of the photons from the metal surface. While the fluorescence enhancement in the case of matching the QD *emission* and plasmon resonances is striking, we can also understand the partial enhancement in the “non-resonant” case (especially the green trace of the 80-200 nm sample) due to the enhancement of the effective *absorption* cross section at 532 nm induced by strong local field of excited surface plasmons. Indeed, by studying the reflectance lineshapes of the various patterned and unpatterned structures (Fig 2 (c)), with and without the quantum dots, we have acquired additional direct evidence indicative of a strong coupling between the plasmons and excitons in the nanocomposites. This coupling is responsible in the dramatic modification in the QD exciton spontaneous emission rate as reflected in the observed enhancement in the fluorescence. More details on the plasmon induced modification of the spontaneous emission is obtained by time-resolved fluorescence lifetime experiments and near-field optical microscopy experiments now under way.

IV. PROBLEMS ENCOUNTERED

- Synthesis of GaN nanowires by MOCVD: Most of the III-N nanowires are synthesized by tube-furnace based VLS growth technique. This process has a wide parametrical window since it is intrinsically close to equilibrium and the anisotropic growth of nanowires is facilitated through the presence of nano-size catalyst particles

or droplets. MOCVD, on the other hand, is typically operated under high supersaturation, which implies that non-selective growth can take place and frustrates the nucleation of nanowires. In order to promote the catalytic growth, sufficient surface diffusion is required. It is our findings that the synthesis of GaN and InGaN is highly sensitive to the surface stoichiometry and process condition. The identification of the optimum process window and the retaining of it has been a major challenge.

- Bandgap engineering of nanostructures: Bandgap engineering using semiconductor alloyed heterostructures has been the hallmarks of modern opto- and microelectronic devices. It is our finding that the synthesis of alloyed nanostructures under near-equilibrium condition poses severe difficulties due to the high strain energy. Traditional synthesis of semiconductor heterostructures is often carried out under non-equilibrium, metastable conditions in which kinetics can be used to counter and even overcome thermodynamic tendency. It is our observation that alloys tend to phase separate spontaneously in either zero- or one-dimensional structures. Creative application of core-shell or coaxial structures with alternating kinetics may provide a solution to this problem.

VI. SIGNIFICANT ACCOMPLISHMENTS in the past 12 month:

- Demonstration of AlGaN coaxial nanowire structures
- Synthesis of InGaN/GaN nanostructures
- Epitaxially alignment of GaN and AlGaN nanowires
- Observation of Stimulated Emission in dense InGaN Nanopost Arrays
- Design and Fabrication of InGaN photonic crystal emitters
- Enhanced fluorescence from coupled quantum dot and plasmonic nanostructures

VII. LIST OF PUBLICATIONS AND PRESENTATIONS

Presentations:

- J. Han, “Frontiers of III-Nitride UV Optoelectronics and Nanostructures”, Rensselaer Polytech Institute, Troy, NY, 2004. **(INVITED)**
- J. Han, “III-Nitride UV Optoelectronics and Nanostructures for Solid State Lighting”, 3rd international workshop on Opto Semiconductor Technologies for Solid State Lighting, Gwangju, Korea, December 2004 **(INVITED)**
- A. Nurmikko, “GaN based Nanostructures for light emitter applications”, Akasaki Research Symposium, Nagoya University, December 2004 **(INVITED)**
- A. Nurmikko, “Physics of Light Emission from III-nitride periodic nanostructures”, International Workshop on Semiconductor Nitrides, Meijo University, Japan, December 2004 **(INVITED)**
- A.V. Nurmikko, Y.-K. Song, and J.-H. Song, Jung Han, “Control of Spontaneous and Stimulated Emission in Wide Bandgap Semiconductor Nanoarrays”, Symposium on Frontiers of Nanoscience, Salt Lake City, April 2005 **(INVITED)**
- J. Han, “III-Nitride UV Optoelectronics and Nanostructures”, Tokyo Institute of Technology, Tokyo, Japan, December 2004.

- J. Han, “III-Nitride UV Optoelectronics and Nanostructures”, Tohoku University, Sendai, Japan, December 2004.
- J. Su, M. Gherasimova, G. Cui, and J. Han, S. Lim, D. Ciuparu, L. Pfefferle, Y. He, A. V. Nurmikko, C. Broadbridge, A. Lehman, “VLS growth of III-Nitride nanowires and heterostructures by MOCVD”, Fall 2004 Meeting of the MRS, Boston, MA, November 2004
- J. Su, M. Gherasimova, G. Cui, and J. Han, H. Peng, E. Makarona, Y. He, Y.-K. Song, and A. V. Nurmikko, “Aligned GaN Nanowires and Nanobridges by MOCVD”, New Haven, CT, March 2005
- Y. He, Y.-K. Song, A.V. Nurmikko, S.-R. Jeon, Z. Ren, M. Gherasimova, J. Han, “Stimulated Emission in the Blue from Dense Arrays of InGaN MQWs Nanoposts, Conference on Lasers and Optoelectronics (CLEO), San Francisco, May
- J. Su, M. Gherasimova, G. Cui, and J. Han, H. Peng, E. Makarona, Y. He, Y.-K. Song, and A. V. Nurmikko, “Aligned GaN Nanowires and Nanobridges by MOCVD”, Electronic Materials Conference, Santa Barbara, CA, June 2005.

Publications:

- M. Gherasimova, J. Su, G. Cui, S.-R. Jeon, Z. Ren, J. Han, Y. He, Y. K. Song, Nurmikko, A.-V.D. Ciuparu, and L. Pfefferle, “A nanocluster route to zero- and one-dimensional quantum structures by MOCVD”, *Phys. Stat. Sol.(a)* (in press)
- J. Su, G. Cui, M. Gherasimova, H. Tsukamoto, J. Han, D. Ciuparu, S. Lim, L. Pfefferle Y. He, A. V. Nurmikko C. Broadbridge, and A. Lehman, “Catalytic growth of GaN Nanowires and Nanostructures by Metal-Organic Chemical Vapor Deposition”, *Appl. Phys. Lett.*, 86, 013105 (2005)
- J. Su, M. Gherasimova, G. Cui, H. Tsukamoto, J. Han, T. Onuma, M. Kurimoto, S. F. Chichibu, C. Broadbridge, A. Lehman, Y. He, and A. V. Nurmikko, “Growth of AlGaIn nanowires by Metal-Organic Chemical Vapor Deposition”, *Appl. Phys. Lett.* (in press)
- “Versatile ultraviolet light emitting diodes for sensor applications”, Yoon-Kyu Song, A. V. Nurmikko, Maria Gherasimova, Seong-Ran Jeon, and Jung Han, *Phys. Stat. Sol. (a)* **201**, 2721 (2004)
- “Fabrication and Performance of Efficient Blue Light Emitting III-Nitride Photonic Crystals”, Lu Chen and A.V. Nurmikko, *Appl. Phys. Lett.* **85**, 3663 (2004)
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- “Optical Properties of Sub-100nm Diameter Nanoposts with Embedded InGaIn Quantum Well Heterostructures”, Yiping He, Lu Chen, Y.-K. Song, and A.V. Nurmikko, S.-R. Jeon, Z. Ren, M. Gherasimova, and J. Han, *Phys. Stat. Sol. (c)* **2**, 2740 (2005)
- “Large enhancement of fluorescence efficiency from CdSe/ZnS quantum dots induced by resonant coupling to spatially controlled surface plasmons”, J.H Song, T. Atay, S. Shi, H. Urabe, and A.V. Nurmikko, *Nanoletters* **5**, 1557 (2005)

VIII. APPROACHES TO BE TAKEN IN THE FOLLOWING YEAR

In the next 12 month contract period we will:

- Develop the InGaN and AlGaIn quantum dot synthesis to create ordered high density arrays and incorporate these to blue/NUV light emitter structures
- Advance the synthesis of InGaIn core-shell and other nanoscale bandgap engineered heterostructures, and evaluate these in close synergy with high spatial resolution spectroscopic optical imaging tools
- Insert these new dense and self-assembled quantum wire nanoscale arrays into basic light emitting diode structures for measurement of device performance.
- Imbed the lithographically fabricated nanopost arrays into LED-type devices and develop a process technology for electrical injection
- Investigate the feasibility of using the recently observed stimulated emission from InGaIn QW nanopost arrays as an enabler for a new generation of laser devices at short visible wavelengths.
- Investigate the use of conducting polymer hosts as a means for electrical contacting to the nitride light emitting nanoparticles

IX. CONCLUSION

We have reported on research results in this project which synergize advanced material science approaches with fundamental optical physics concepts pertaining to light-matter interaction, with the goal of solving seminal problems for the development of very high performance light emitting diodes (LEDs) in the blue and near ultraviolet for Solid State Lighting applications. Accomplishments in the first 12 month contract period include (i) new means of synthesizing zero- and one-dimensional GaN nanostructures, (ii) establishment of the building blocks for making GaN-based microcavity devices, and (iii) demonstration of top-down approach to nano-scale photonic devices for enhanced spontaneous emission and light extraction. These include a demonstration of eight-fold enhancement of the external emission efficiency in new InGa_N QW photonic crystal structures. The body of results is presented in this report shows how a solid foundation has been laid, with several noticeable accomplishments, for innovative research, consistent with the stated milestones.

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- ²⁷ GaN wafers with a dimension of 1x1 cm² is employed. Pieces of wafers are placed on a SiC coated graphite susceptor having a diameter of 3 inches.
- ²⁸ Coltrin et al., J. Cryst. Growth (2001)

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DE-FC26-03NT41941

Name & Phone No. of DOE COR

Dr. Joel Chaddock, 304-285-0958

☒ For Technical Papers/Journal Articles/Presentations
Mark P. Dvorscak
U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Frontiers of III-Nitride UV Optoelectronics and Nanostructures

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation
☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆ 4. Results of Review for Possible Inventive Subject Matter:

- a. ☒ No Subject Invention is believed to be disclosed therein.
- b. ☐ Describes a possible Subject Invention relating to _____
- i. Awardee Docket No.: _____
- ii. A disclosure of the invention was submitted on _____
- iii. A disclosure of the invention will be submitted by the following date: _____
- iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or
☐ will be applied for by the following date: _____

◆ 5. Signed  Date 8/29/05
(Awardee)

Name & Phone No. Prof. Jung Han, 203-432-7567

Address Yale Univ., Dept of EE, 15 Prospect St., PO Box 208284, New Haven, CT 06520

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

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U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. AWARDEE ACTION (AWARDEE COMPLETES PART A. 1-5)

1. Document Title: III-Nitride UV Optoelectronics and Nanostructures for Solide State Lig

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

ii. A disclosure of the invention was submitted on _____

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B. DOE PATENT COUNSEL ACTION

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. AWARDEE ACTION (AWARDEE COMPLETES PART A. 1-5)

1. Document Title: GaN based Nanostructures for light emitter applications
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
- ◆4. Results of Review for Possible Inventive Subject Matter:
 - a. ☒ No Subject Invention is believed to be disclosed therein.
 - b. ☐ Describes a possible Subject Invention relating to _____
 - i. Awardee Docket No.: _____
 - ii. A disclosure of the invention was submitted on _____
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☐ will be applied for by the following date: _____

◆5. Signed  Date 8/29/05
(Awardee)

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

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B. DOE PATENT COUNSEL ACTION

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

◆ **Must be completed by the awardee.**

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Physics of Light Emission from III-nitride periodic nanostructures

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation
☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆ 4. Results of Review for Possible Inventive Subject Matter:

- a. ☒ No Subject Invention is believed to be disclosed therein.
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- i. Awardee Docket No.: _____
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☐ will be applied for by the following date: _____

◆ 5. Signed Arto Nurmi (Awardee) Date 8/29/05

Name & Phone No. Prof. Arto Nurmi, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

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U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Control of Spontaneous and Stimulated Emission in Wide Bandgap Semicon

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

ii. A disclosure of the invention was submitted on _____

iii. A disclosure of the invention will be submitted by the following date: _____

iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or

☐ will be applied for by the following date: _____

◆5. Signed Arto Nurmikko Date 8/29/05
(Awardee)

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

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◆ Award No.

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Name & Phone No. of DOE COR

Dr. Joel Chaddock, 304-285-0958

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: VLS growth of III-Nitride nanowires and heterostructures by MOCVD
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
- ◆ 4. Results of Review for Possible Inventive Subject Matter:
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 - b. ☐ Describes a possible Subject Invention relating to _____
 - i. Awardee Docket No.: _____
 - ii. A disclosure of the invention was submitted on _____
 - iii. A disclosure of the invention will be submitted by the following date: _____
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☐ will be applied for by the following date: _____

◆ 5. Signed  Date 8/29/05
(Awardee)

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

- ☐ Patent clearance for release of the above-identified document is granted.
☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Aligned GaN Nanowires and Nanobridges by MOCVD
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
- ◆ 4. Results of Review for Possible Inventive Subject Matter:
 - a. ☒ No Subject Invention is believed to be disclosed therein.
 - b. ☐ Describes a possible Subject Invention relating to _____
 - i. Awardee Docket No.: _____
 - ii. A disclosure of the invention was submitted on _____
 - iii. A disclosure of the invention will be submitted by the following date: _____
 - iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or
☐ will be applied for by the following date: _____

◆ 5. Signed Arto Nurmikko (Awardee) Date 8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

- ☐ Patent clearance for release of the above-identified document is granted.
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Signed _____ Date _____
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U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Stimulated Emission in the Blue from Dense Arrays of InGaN MQWs Nanopo

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☐ Journal Article ☒ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆ 4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

ii. A disclosure of the invention was submitted on _____

iii. A disclosure of the invention will be submitted by the following date: _____

iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or
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◆ 5. Signed _____

(Awardee)

Date _____

8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____

(Patent Attorney)

Date _____

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U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. AWARDEE ACTION (AWARDEE COMPLETES PART A. 1-5)

1. Document Title: A nanocluster route to zero- and one- dimensional quantum structures
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
- ◆4. Results of Review for Possible Inventive Subject Matter:
 - a. ☒ No Subject Invention is believed to be disclosed therein.
 - b. ☐ Describes a possible Subject Invention relating to _____
 - i. Awardee Docket No.: _____
 - ii. A disclosure of the invention was submitted on _____
 - iii. A disclosure of the invention will be submitted by the following date: _____
 - iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or
☐ will be applied for by the following date: _____

◆5. Signed _____

(Awardee)

Date _____

8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE PATENT COUNSEL ACTION

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____

(Patent Attorney)

Date _____

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Catalytic growth of GaN Nanowires and Nanostructures by Metal-Organic

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆ 4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

ii. A disclosure of the invention was submitted on _____

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Date

8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

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U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Growth of AlGaIn Nanowires by Metal-Organic Chemical Vapor Deposition

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆ 4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

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iii. A disclosure of the invention will be submitted by the following date: _____

iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or

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Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

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B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Versatile ultraviolet light emitting diodes for sensor applications
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
- ◆4. Results of Review for Possible Inventive Subject Matter:
 - a. ☒ No Subject Invention is believed to be disclosed therein.
 - b. ☐ Describes a possible Subject Invention relating to _____
 - i. Awardee Docket No.: _____
 - ii. A disclosure of the invention was submitted on _____
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B. DOE Patent Counsel Action

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Name & Phone No. of DOE COR

Dr. Joel Chaddock, 304-285-0958

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Fabrication and Performance of Efficient Blue Light Emitting III-Nitride
2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation
☐ Other (please specify) _____
3. Date Clearance Needed: _____
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◆ 5. Signed


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Date

8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

- ☐ Patent clearance for release of the above-identified document is granted.
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A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Epitaxial growth of aligned AlGaInN nanowires by Metal-Organic Chemical Vapor Deposition

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

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(Patent Attorney)

◆ Must be completed by the awardee.

REQUEST FOR PATENT CLEARANCE FOR RELEASE OF CONTRACTED RESEARCH DOCUMENTS

TO: ☐ For Technical Reports
AAD Document Control
MS 921-143
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◆ Award No.

DE-FC26-03NT41941

Name & Phone No. of DOE COR

Dr. Joel Chaddock, 304-285-0958

☒ For Technical Papers/Journal Articles/Presentations
Mark P. Dvorscak
U.S. Department of Energy
9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Optical Properties of Sub-100nm Diameter Nanoposts with Embedded InGaN

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report

☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation

☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆4. Results of Review for Possible Inventive Subject Matter:

a. ☒ No Subject Invention is believed to be disclosed therein.

b. ☐ Describes a possible Subject Invention relating to _____

i. Awardee Docket No.: _____

ii. A disclosure of the invention was submitted on _____

iii. A disclosure of the invention will be submitted by the following date: _____

iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or

☐ will be applied for by the following date: _____

◆5. Signed _____

(Awardee)

Date

8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

☐ Patent clearance for release of the above-identified document is granted.

☐ Other: _____

Signed _____

(Patent Attorney)

Date _____

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9800 S. Cass Avenue
Argonne, IL 60439
FAX: (630) 252-2779

A. Awardee Action (Awardee Completes Part A. 1-5)

1. Document Title: Large enhancement of fluorescence efficiency from CdSe/ZnS quantum dot

2. Type of Document: ☐ Technical Progress Report ☐ Topical Report ☐ Final Technical Report
☐ Abstract ☐ Technical Paper ☒ Journal Article ☐ Conference Presentation
☐ Other (please specify) _____

3. Date Clearance Needed: _____

◆4. Results of Review for Possible Inventive Subject Matter:

- a. ☒ No Subject Invention is believed to be disclosed therein.
- b. ☐ Describes a possible Subject Invention relating to _____
- i. Awardee Docket No.: _____
- ii. A disclosure of the invention was submitted on _____
- iii. A disclosure of the invention will be submitted by the following date: _____
- iv. A waiver of DOE's patent rights to the awardee: ☐ has been granted, ☐ has been applied for, or
☐ will be applied for by the following date: _____

◆5. Signed Arto (Awardee) Date 8/29/05

Name & Phone No. Prof. Arto Nurmikko, 401-863-2869

Address Brown Univ., Div. of Engineering, 182 Hope St., Box D, Providence, RI 02912

B. DOE Patent Counsel Action

- ☐ Patent clearance for release of the above-identified document is granted.
- ☐ Other: _____

Signed _____ Date _____
(Patent Attorney)

◆ Must be completed by the awardee.